

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

THIS PAGE BLANK (USPTO)

(19) Patent Office of Japan (JP) (11) Publication of Patent Application

(12) JAPANESE PATENT REPORT

Showa 62-69202

(51) Int. CL. 5 ID Code Office Cont'l No. (43) Publication: Showa 62 (1987) 3/30

G 02 B 5/30 7529-2H

Verification request: not requested

Number of claims of the invention: 1

Number of pages (total of 5)

(54) Name of the invention: Light Polarization Film

(21) Filed Number: Showa 60-209802

(22) Filed Date: Showa 60 (1985) 9/20

Patent Assignee: Fuji Photo Film Company

JP 62-69202

Note: Names, addresses, company names and brand names are translated in the most common manner. Japanese language does not have singular or plural words unless otherwise specified by a numeral prefix or a general form of plurality suffix.

Description of the invention

1. Name of the invention

Light Polarization Film

2. Scope of the claims

A light polarization film characterized by the fact that it is a material where inside a hydrophobic polymeric film, that is obtained from a liquid crystal state as it has been uniaxially extended in a liquid state or by spreading a molten flow, a dichroic dye material is oriented.

3. Detailed explanation of the invention

[Field of the invention]

The present invention is an invention about a light polarization film. The light polarization film is used in the field of electric trains, or clocks (watches) etc., and besides that it is also widely used in automobile meters, different types of metering devices. Together with the wide use in such fields, the requirements relative to the properties necessary in the polarization films, especially, the humidity resistance properties, the heat resistance properties, have increased.

[Previous technology]

At the present time, the usually used light polarization film is a material where in polyvinyl alcohol (here below called PVA), iodine or a dichroic dye material is incorporated and then it is uniaxially oriented, and when it is placed in a high temperature or high humidity ambient environment, because

of the thermal contraction and the moisture absorption properties of the PVA, an orientation relaxation occurs and the light polarization properties are decreased, and there is a color change, and a material that sufficiently satisfies the requirements, is not obtained.

In order to improve upon this drawback point, there is the material where the protective layer of the light polarization film is improved (for example, Japanese Patent Application Laid Open Number Showa 57-118220, Japanese Patent Application Laid Open Number Showa 57-30808, Japanese Patent Application Laid Open Number Showa 56-80001), or the material where the PVA of the light polarization film has been improved, the material where instead of the PVA another polymer materials is used (for example, Japanese Patent Application Laid Open Number Showa 56-25419, Japanese Patent Application Laid Open Number Showa 57-115507, Japanese Patent Application Laid Open Number Showa 54-45153, Japanese Patent Application Laid Open Number Showa 54-850050, Japanese Patent Application Laid Open Number Showa 55-87112, Japanese Patent Application Laid Open Number Showa 55-135808, Japanese Patent Application Laid Open Number Showa 56-125701), etc. However, the humidity resistance properties, which were the goal, were not necessarily sufficiently improved, and the circumstances were such that it was difficult to obtain materials that had sufficiently large degree of the light polarization.

[Goal of the present invention]

The present invention is an invention that suggests a light polarization film that has improved humidity resistance properties, which represented the above described drawback point.

[Structure of the invention]

According to the present invention, it was understood that by orienting a dichroic dye material in a hydrophobic polymeric film that has been manufactured from a liquid state, it is possible to obtain a light polarization film that has a large light polarization degree and also that has humidity resistance properties and heat resistance properties.

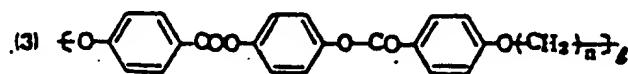
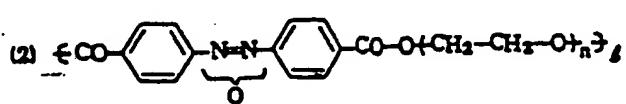
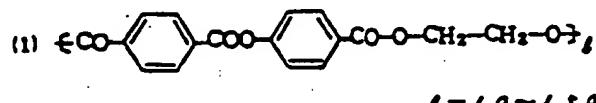
According to the present invention, in a liotropic polymeric liquid crystal or semi-liotropic polymeric liquid crystal a dichroic dye material is mixed, and the film is manufactured, or the film manufactured from the above described

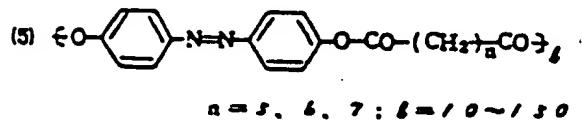
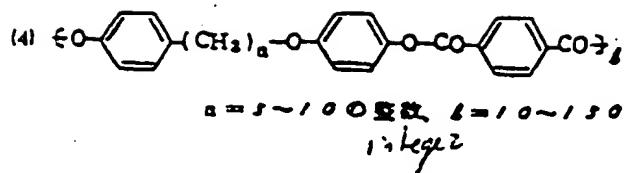
polymeric liquid crystal is immersed in a dichroic dye material solution, or a solution of the dichroic dye material is coated, or on the film that is manufactured from the liquid crystal state a dichroic dye material is vapor deposited, and it was understood that the goal was achieved by using any of these methods.

As the used polymeric material, a material is used that has practically no solubility properties relative to water, and also, shows liotropic liquid crystal properties in the solution state, or shows semi-liotropic properties upon heating.

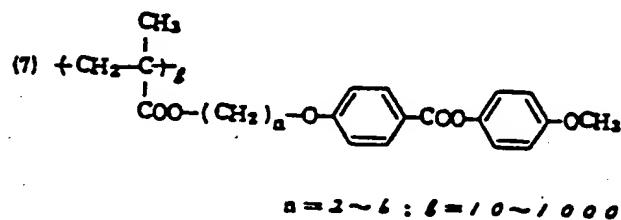
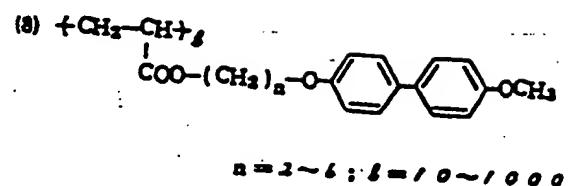
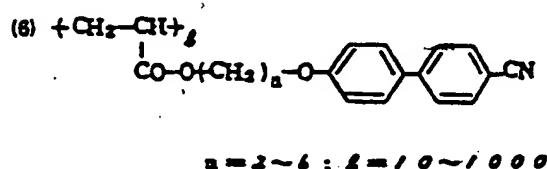
As the materials that show liotropic liquid crystal properties, it is a solution, which is a polymeric solution that displays anisotropic properties above a certain concentration. For example, there are the methylene chloride solution, the chloroform solution of ethyl cellulose, the cellulose acetate butyrate and acetone - methanol mixture (4:1) solution, the cellulose acetate dioxane solution, the cellulose triacetate dichloroacetic acid solution etc., and especially, a large number of examples of cellulose derivative materials have been reported in Colloid and Polymer Sci. 258, 1335 ~ 1342 (1980), in the Japanese Patent Application Laid Open Number Showa 58-96230, etc. The polymethyl - L - glutamate chloroform solution, the methylene dichloride solution, the polybenzyl - L - glutamate methylene dichloride solution, dioxane solution, choloroform solution, etc., also are materials that show liquid crystal properties, and these have been reported in details in Table 18, pages 705 ~ 723 (1980), etc.

As the semi-liotropic liquid crystals, the materials can be classified into materials that contain a mesogenic radical in the main chain and materials that contain a mesogenic radical in the side chain. As examples of the former type, for example, there are the following:





As examples of the latter type, there are the following:



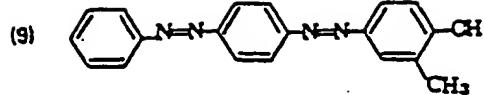
These and other materials can be used and these have been described especially in details in Table 26, pages 479 ~ 491 (1982).

As the dichroic dye material, there are the direct dye materials that display dichroic properties, the basic properties possessing dye materials, the acidic properties possessing dye materials, the dispersed dye materials, the host type dye materials that are used in the guest - host liquid crystals, etc.

For example, the dye materials shown on the separate pages, etc., materials can be used.

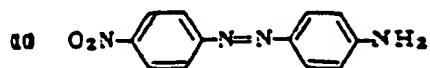
And these dye materials can be used individually or also as mixtures.

The method for the formation of polymeric films usually can be classified into the method where a solution flow flows and is spread in a liquid state over a supporting (substrate) material and the method where it is extruded in a molten state. And in the case of the present invention there is no problem in using any of these types of methods; and if at the time of the flow spreading or the extrusion, a constant (certain) shear stress is applied, the polymeric liquid crystal is oriented in that direction. In the case of a nematic liquid crystal, immediately after the spreading the solvent agent is eliminated, and in the case of the semi-nematic liquid crystal, after the

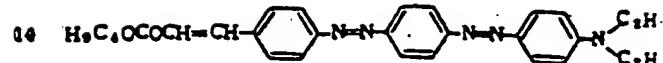
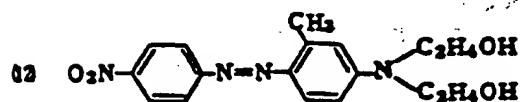


(9)

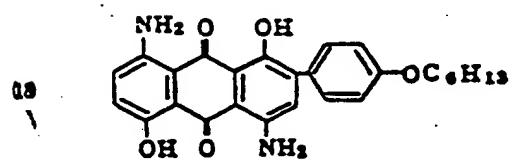
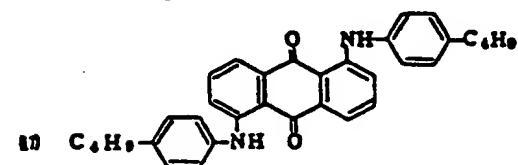
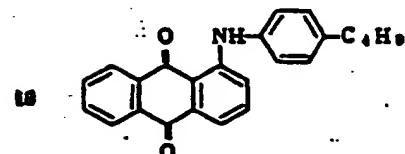
特開昭62-69202(3)



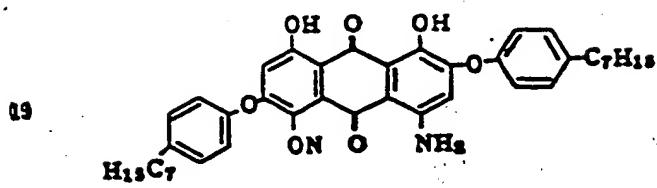
(1) $O_2N-C_6H_4-N=N-C_6H_4-N=N-C_6H_4$



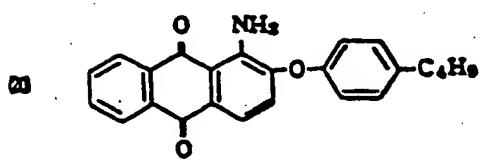
19 



18



g9



20



extrusion, it is quickly cooled off, and by that, the polymeric liquid crystal is fixed in the oriented state as it is. If at this time the dichroic dye material is mixed together and the dye material is oriented together with the polymeric material, it displays light polarization properties, and also, if on the oriented film material dichroic dye material is vapor deposited or if a dichroic dye material is adhered and the film is dyed after the orientation of the molecules of the film, it is possible to obtain a material that shows light polarization properties.

In the case of the usual light polarization film material, usually, isotropic properties possessing film is manufactured, and then this film is subjected to uniaxial orientation, however, in the case of such orientation (extension), because of the fact that the orientation coefficient is high, there is a residual orientation distortion, and it is a condition where if the temperature is increased, moisture is absorbed, and the orientation returns to the initial state (orientation relaxation). However, according to the present invention, in the case when a film is manufactured as a flow is spread as it is in a liquid state, there are no residual distortions, and there is no generation of orientation relaxation, and it is possible to obtain light polarization film that has durability properties.

It is also possible to add lubricant agents, etc., in the polymeric liquid crystal solution as long as they do not destroy the liquid crystal properties, and there are no problems.

Here below, practical implementation examples will be reported, and the describe din these examples, measurements and evaluation standards are according to the presented here below.

(1) Birefringence (birefraction)

The retardation of the sample was measured by using a white color light beam and a polarized light microscope with an attached compensator, and this measured value was then divided by the film thickness and the birefraction was obtained.

(2) Humidity resistance properties of the light polarization film

The film was placed for a period of 5 days in a 60°C x 80 % RH environment, and after that the degree of the light polarization was

measured. The materials where the decrease of the light polarization was within 10 % of the initial degree of light polarization, were denoted as A, the materials where the decrease was in the range of 10 ~ 30 %, were denoted as B, and the materials where it was decreased by more than 30 % were denoted as C materials.

(3) Heat resistance properties

The film was placed for a period of 5 days in a 100°C dry environment, and after that the degree of the light polarization was measured. The materials where the decrease of the light polarization was within 10 % of the initial degree of light polarization, were denoted as A, the materials where the decrease was in the range of 10 ~ 30 %, were denoted as B, and the materials where it was decreased by more than 30 % were denoted as C materials.

(4) Measurement of the light polarization degree

The light polarization degree was measured by using a spectrophotometer and measuring the spectral acceleration coefficient in the visible region (400 ~ 780 nm wavelength). It is calculated from the light beam transmittance at the maximum absorption wavelength when two pieces of the light polarization film are placed so that their orientation axes are mutually parallel (T_{parallel}), and from the light beam transmittance at the maximum absorption wavelength when two pieces of the light polarization film are placed so that their orientation axes are mutually perpendicular ($T_{\text{perpendicular}}$).

$$\text{Light polarization degree (\%)} = \frac{T_{\text{parallel}} - T_{\text{perpendicular}}}{T_{\text{parallel}} + T_{\text{perpendicular}}} \times 100$$

Practical Example 1

A 25 % chloroform solution of polybutyl-L-glutamate (PBLG) was prepared.

This solution showed a rainbow color specific to the cholesteric liquid crystal, and it was in a liquid crystal state.

By using a doctor blade, this was coated on the surface of a glass plate so that the dried thickness became 10 microns, and it was dried at a temperature of 30°C for a period of 5 minutes and after that especially, it was dried at a temperature of 80°C for a period of 5 minutes, and by that the PBLG layer was obtained.

The birefraction value was $2.3 \times 10 - 2$.

On this film the compound (T2) which is an azo type dye material was vacuum vapor deposited, and on both sides, as a protective layer a 125 micron cellulose triacetate film material was laminated, and by that the light polarization film material was obtained.

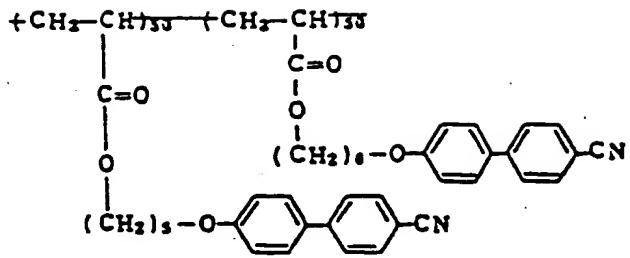
The light polarization degree and the durability properties of the obtained light polarization film material were shown according to Table 1, and as it is seen they were materials that showed excellent light polarization properties and also good humidity resistance and temperature resistance properties.

Practical Example 2

45 % methylene chloride solution of ethyl cellulose was prepared. This solution presented a cholesteric color. In this solution the compound (10) was added in an amount of 2.5 % relative to the ethyl cellulose. By using a doctor blade, this solution was coated on the surface of a glass plate so that the dried thickness became 8 microns, and it was dried at a temperature of 30°C for a period of 5 minutes, and especially, it was dried at a temperature of 60°C for a period of 5 minutes. The birefraction of the obtained film material was $1.5 \times 10 - 2$. And especially, on both sides of this film, as a protective film 125 micron cellulose triacetate was laminated and by that the light polarization film material was obtained. As it is shown according to the presented in Table 1, a light polarization film material was obtained that has humidity resistance properties and temperature resistance properties.

Practical Example 3

A copolymer material with the following here below composition was synthesized.



To this copolymer material the compound (17) dichroic dye material was added in an amount of 1.5 % relative to the polymer material, and at a temperature of 130°C, a shear stress was applied so that it was melt extruded on the surface of a glass plate and it became 20 microns.

The birefraction of the obtained film material was 0.15. And especially, on both sides of this film, as a protective film 125 micron cellulose triacetate was laminated and by that the light polarization film material was obtained. As it is shown according to the presented in Table 1, a film material was obtained that has good ;light polarization properties, humidity resistance properties and temperature resistance properties.

Reference Example

A 75 micron thick polyvinyl alcohol film material was immersed for 1 minute in a solution containing 2 weight % iodine, 4 weight % potassium iodide, and after that this was oriented at an orientation ratio of 4 times, and after that it was immersed for a period of 1 minute in a solution containing 4 weight % of boric acid, and then it was dried at a temperature of 100°C for a period of 10 minutes. On both sides of this film cellulose triacetate was laminated, and a light polarization film material, was obtained. As it is shown according to the presented in Table 1, the initial degree of light polarization was good, however, the humidity resistance properties were completely insufficient.

Table 1

	初期偏光度	耐湿性	耐温性	
実施例1	95%	A	A	3
実施例2	80%	A	A	4
実施例3	85%	A	A	5
比較例	77%	C	B	2

Headings in the table:

1. Practical Examples, 2. Reference Examples, 3. initial light polarization degree, 4. humidity resistance properties, 5. humidity resistance properties (probably an error and one of the columns should be temperature resistance, but it is not possible to know which one – translator's note).

Patent Assignee: Fuji Photo Film Company

Translated by Albena Blagev ((651) 735-1461 (h), (651) 704-7946 (w))

11/29/99

THIS PAGE BLANK (USPTO)